REPORT DOCUMENTATION PAGE

Form Approved OMB NO. 0704-0188

1. REPORT DATE (DD-MM-YYYY)	2. REPORT TYPE	3. DATES COVERED (From - To)
30-07-2015	Final Report	1-Oct-2014 - 30-Jun-2015

4. TITLE AND SUBTITLE
Final Report: Atomically Accurate Structure Analysis for InAs / InAsSb Strained–Layer Superlattices STIR, Dr. William Clark, Electronics Division

5a. CONTRACT NUMBER
W911NF-14-1-0645

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER
611102

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

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8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS (ES)
U.S. Army Research Office
P.O. Box 12211
Research Triangle Park, NC 27709-2211

10. SPONSOR/MONITOR'S ACRONYM(S)
ARO

11. SPONSOR/MONITOR'S REPORT NUMBER(S)
66420-EL-II.2

12. DISTRIBUTION AVAILIBILITY STATEMENT
Approved for Public Release; Distribution Unlimited

13. SUPPLEMENTARY NOTES
The views, opinions and/or findings contained in this report are those of the author(s) and should not contrued as an official Department of the Army position, policy or decision, unless so designated by other documentation.

14. ABSTRACT
We use cross-sectional STM to effectively discriminate between arsenic and antimony sites in mixed group–V semiconductor alloys grown by MBE. Our effort is directed toward two problems representing distinctly different facets of a common material system: InAsSb. Project 1, undertaken jointly with Sandia National Laboratories, assesses the difference between the as–grown and intended [001] antimony profile in an InAs / InAsSb strained–layer superlattice arising from antimony segregation and cross-incorporation. We show how the STM data fully explain the observed HRXRD spectrum and predict a corresponding strain profile that may also be tested against

15. SUBJECT TERMS
cross–sectional scanning tunneling microscopy, InAs / InAsSb superlattices, metamorphic bulk InAsSb alloys

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	15. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			Michael Weimer
UU	UU	UU	UU		19b. TELEPHONE NUMBER
					979-458-7909

Report Title

Final Report: Atomically Accurate Structure Analysis for InAs / InAsSb Strained–Layer Superlattices STIR, Dr. William Clark, Electronics Division

ABSTRACT

We use cross-sectional STM to effectively discriminate between arsenic and antimony sites in mixed group–V semiconductor alloys grown by MBE. Our effort is directed toward two problems representing distinctly different facets of a common material system: InAsSb. Project 1, undertaken jointly with Sandia National Laboratories, assesses the difference between the as–grown and intended [001] antimony profile in an InAs / InAsSb strained–layer superlattice arising from antimony segregation and cross-incorporation. We show how the STM data fully explain the observed HRXRD spectrum and predict a corresponding strain profile that may also be tested against TEM. Project 2, undertaken jointly with the Army Research Laboratory, addresses the anion sublattice order in a bulk, metamorphic InAsSb alloy. We demonstrate how cross–sectional STM permits a semi-quantitative assessment of short–range order by way of an image–based antimony–antimony correlation function reconstructed from large–area STM surveys. Project 1 has a direct impact on the predicted band structure and carrier transport in MWIR strained–layer superlattices, whereas Project 2 potentially bears on the ordering–induced band–gap anomaly in LWIR bulk alloys.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

Received		Paper
07/27/2015	1.00	M.R. Wood, K. Kanedy, F. Lopez, M. Weimer, J.F. Klem, S.D. Hawkins, E.A. Shaner, J.K. Kim. Monolayer-by-monolayer compositional analysis of InAs/InAsSb superlattices with cross-sectional STM, Journal of Crystal Growth, (09 2015): 110. doi:
TOTAL:		**1**

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

Received Paper

TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

Number of Presentations: 0.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Received Paper

TOTAL:

Number of Manuscripts:

Books

Received	Book

TOTAL:

Received	Book Chapter

TOTAL:

Patents Submitted

Patents Awarded

Awards

Graduate Students

NAME	PERCENT_SUPPORTED	Discipline
Kara M. Kanedy	1.00	
Matthew R. Wood	0.36	
FTE Equivalent:	**1.36**	
Total Number:	**2**	

Names of Post Doctorates

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Faculty Supported

NAME	PERCENT_SUPPORTED	National Academy Member
Michael B. Weimer	0.07	
FTE Equivalent:	**0.07**	
Total Number:	**1**	

Names of Under Graduate students supported

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: 0.00

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The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:...... 0.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):...... 0.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:...... 0.00

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The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields:...... 0.00

Names of Personnel receiving masters degrees

NAME
Total Number:

Names of personnel receiving PHDs

NAME
Total Number:

Names of other research staff

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

PROJECT 1

- Monolayer–by–monolayer compositional analysis of InAs /InAsSb superlattices with cross–sectional STM
- Collaboration with Sandia National Laboratories
  J.F. Klem, S.D. Hawkins, E.A. Shaner, and J.K. Kim

Highlights

- device-scale STM surveys are used to study an InAs/InAsSb superlattice grown by MBE
- the as–grown antimony profile differs substantially from design intentions
- this profile is quantitatively explained by segregation and cross incorporation
- the total antimony content per period varies as the growth progresses
- the (004) HRXRD spectrum is completely accounted for by the STM composition profile
- the corresponding strain profile mirrors the composition profile, permitting direct comparison with TEM
- these findings have implications for both band structure and carrier mobility

Publication

- Journal of Crystal Growth, 425, 110–114 (2015)
  UPLOADED

PROJECT 2

- Cross–sectional STM analysis of short–range order in metamorphic InAsSb alloys
- Collaboration with Army Research Laboratory
  S.P. Svensson and W.L. Sarney

Highlights

- a bulk, MBE–grown InAsSb alloy deposited on a metamorphic buffer matched to GaSb has been successfully cleaved in (110) cross section and imaged with STM
- device–scale STM surveys illustrate short–range order on the mixed, group–V sublattice
- the antimony–antimony correlation function obtained from the reciprocal–space power spectral density demonstrates preferred, next–nearest–neighbor antimony–for–arsenic replacement over distances corresponding to several unit cells
- antimony voids are also noted, but their size and spatial distribution have not been quantified

• the band-gap defect associated with bulk ordering is expected to significantly perturb the threshold for optical absorption at wavelengths approaching the far end of the LWIR regime

Technical Figures and Accompanying Explanation

• ATTACHED

Technology Transfer

FIGURE CAPTIONS

FIGURE 1. GROWTH SEQUENCE AND WAFER DIAGRAM

We examined an aluminum–free growth (K1229) as a test of our ability to image a metamorphically grown sample. This sample consisted of a graded InGaSb buffer grown on GaSb substrate. An InGaSb virtual substrate and a unstrained, bulk InAsSb alloy were grown atop the graded buffer. Knowing that concentrations and thicknesses can sometimes be non–uniform at the edges of a growth we chose to look at the sample furthest removed from the edge of the growth.

FIGURE 2. EXPERIMENTAL HRXRD SPECTRUM

The experimental high–resolution x–ray diffraction spectrum shows a relatively sharp peak from the thick bulk alloy and a broad peak due to the thin virtual substrate. Calculations based on approximate mismatches taken directly from the graph confirm the nominal alloy concentrations in FIGURE 1.

FIGURE 3. LARGE–AREA STM SURVEYS

Large–area STM surveys taken over the interface between the virtual substrate and the bulk alloy (left) and approximately a quarter of the way into the growth of the bulk alloy (right). The mixed common–atom, non–common–atom junction shows GaAs interfacial bonds (darker sites) along the interface marked by the caret. Bright sites in both materials identify InSb–like back–bonded surface anions within a matrix of GaSb (virtual substrate) or InAs (bulk alloy).

FIGURE 4. ANION SUBLATTICE ORDER

A representative atomic–resolution image from the bulk alloy survey shown in FIGURE 3. Isovalent antimony–for–arsenic substitutions within the cleavage–exposed (1) and second subsurface (3) planes are denoted by carets. These substitutions appear preferentially situated at next–nearest–neighbor anion sites (encircled in blue) along the [-110] direction. Relatively large voids, where antimony is not present in the cleavage–exposed plane, suggest the presence of longer–range correlations in the distribution of antimony atoms.

(Note: as this was our first experience with this material system, the images – while good – are not up to the high standards we set for ourselves in this lab and as such were gently processed to bring out the atomic corrugation for display purposes. We fully expect to obtain better images as we become more accustomed to this material.)

FIGURE 5. ANION SUBLATTICE ORDER

The correlations in antimony–for–arsenic replacement noted above are seen to be statistically significant by way of the autocorrelation map (left) generated from the ensemble of bulk alloy images and the corresponding section (right) along the [-110] direction between the carets in the map. Vertical marks (right) separated by twice the lattice constant mirror the in–plane ordering of antimony atoms revealed in FIGURE 4. This section also suggests (although qualitatively) that the ordering falls off exponentially with distance from the origin. A discretized model[1], where equally spaced lattice sites are either occupied or unoccupied, and correlations computed from this discretized map are required to quantitatively describe this decay constant.

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[1] See for example: J. Steinshnider, Cross–Sectional Scanning Tunneling Microscopy as a Probe of Local Order in Semiconductor Alloys, A. Mascarenhas (Ed.) Spontaneous Ordering in Semiconductor Alloys

Metamorphic InAsSb

Growth Sequence and Wafer Diagram

growth sequence

$InAsSb_{0.20}$	0.96 μm	bulk alloy
$In_{0.17}GaSb$	0.2 μm	virtual substrate
$In_{0.19}GaSb$ • • • $In_{0.00}GaSb$	1.1 μm	graded buffer
n–GaSb		substrate

wafer diagram

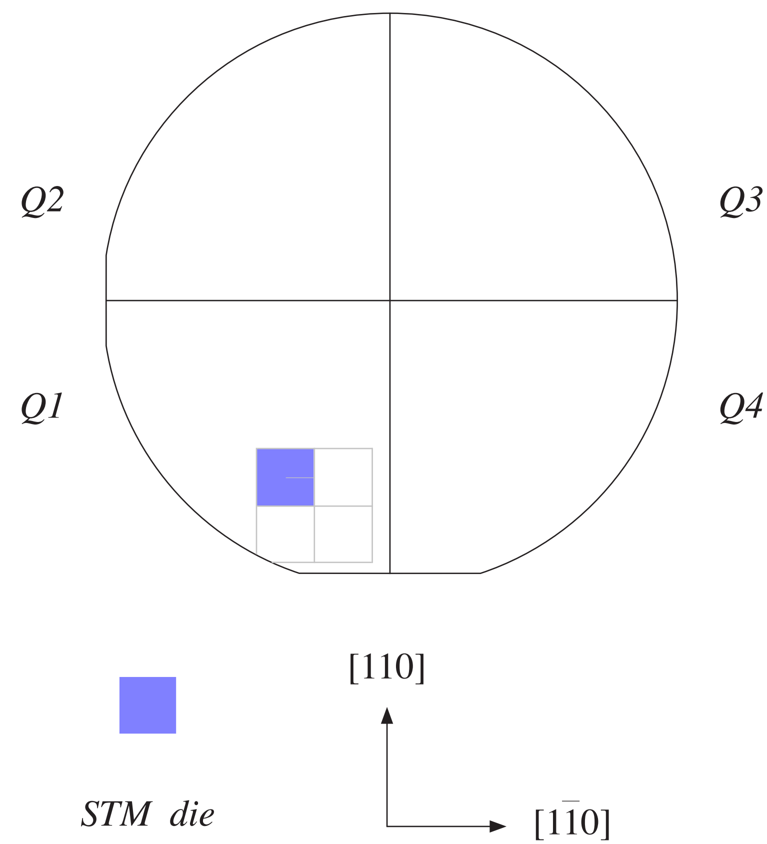

Figure 1

METAMORPHIC InAsSb

Experimental HRXRD Spectrum

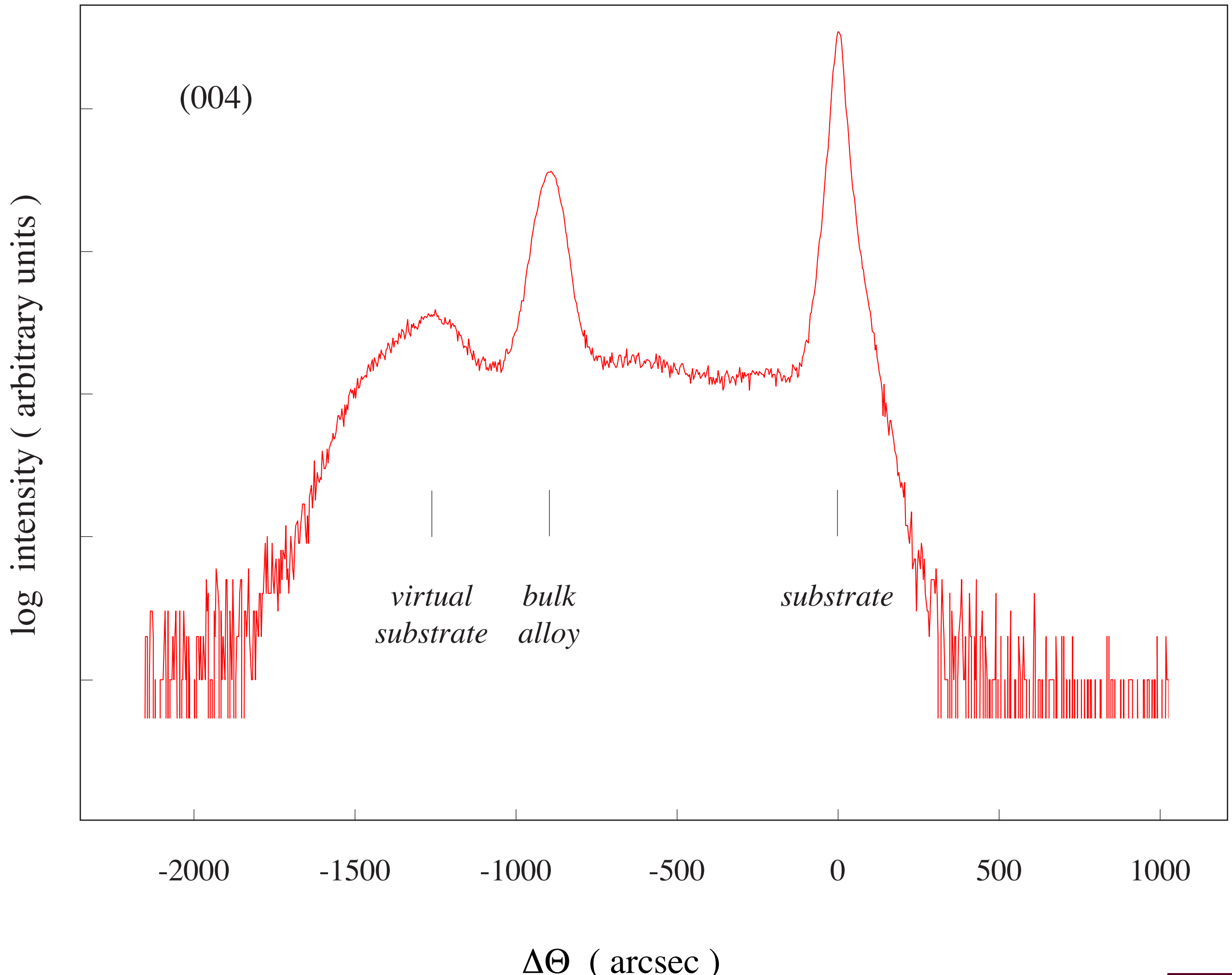

Figure 2

Metamorphic InAsSb

Large–Area STM Surveys

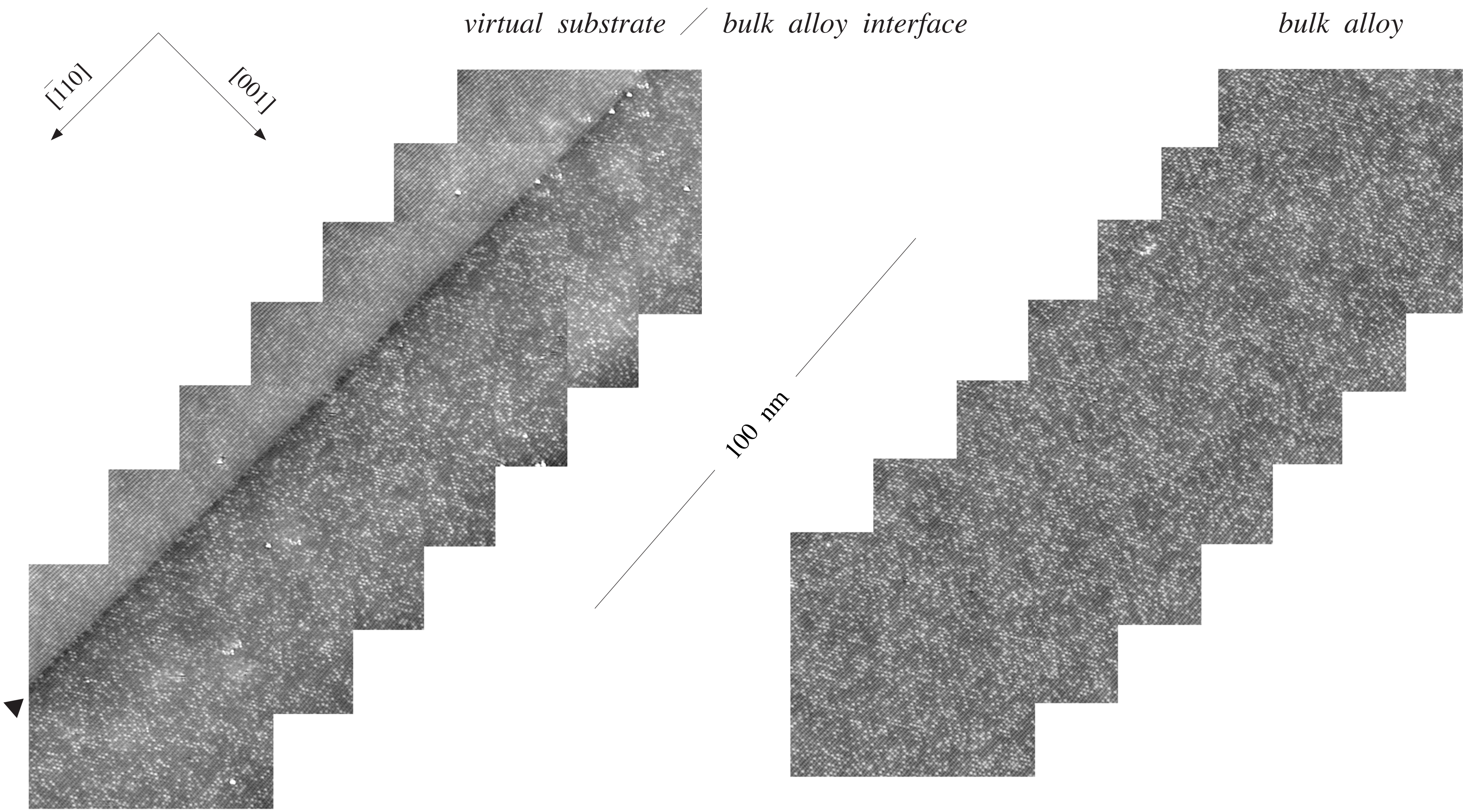

Figure 3

METAMORPHIC INASSB

Anion Sublattice Order

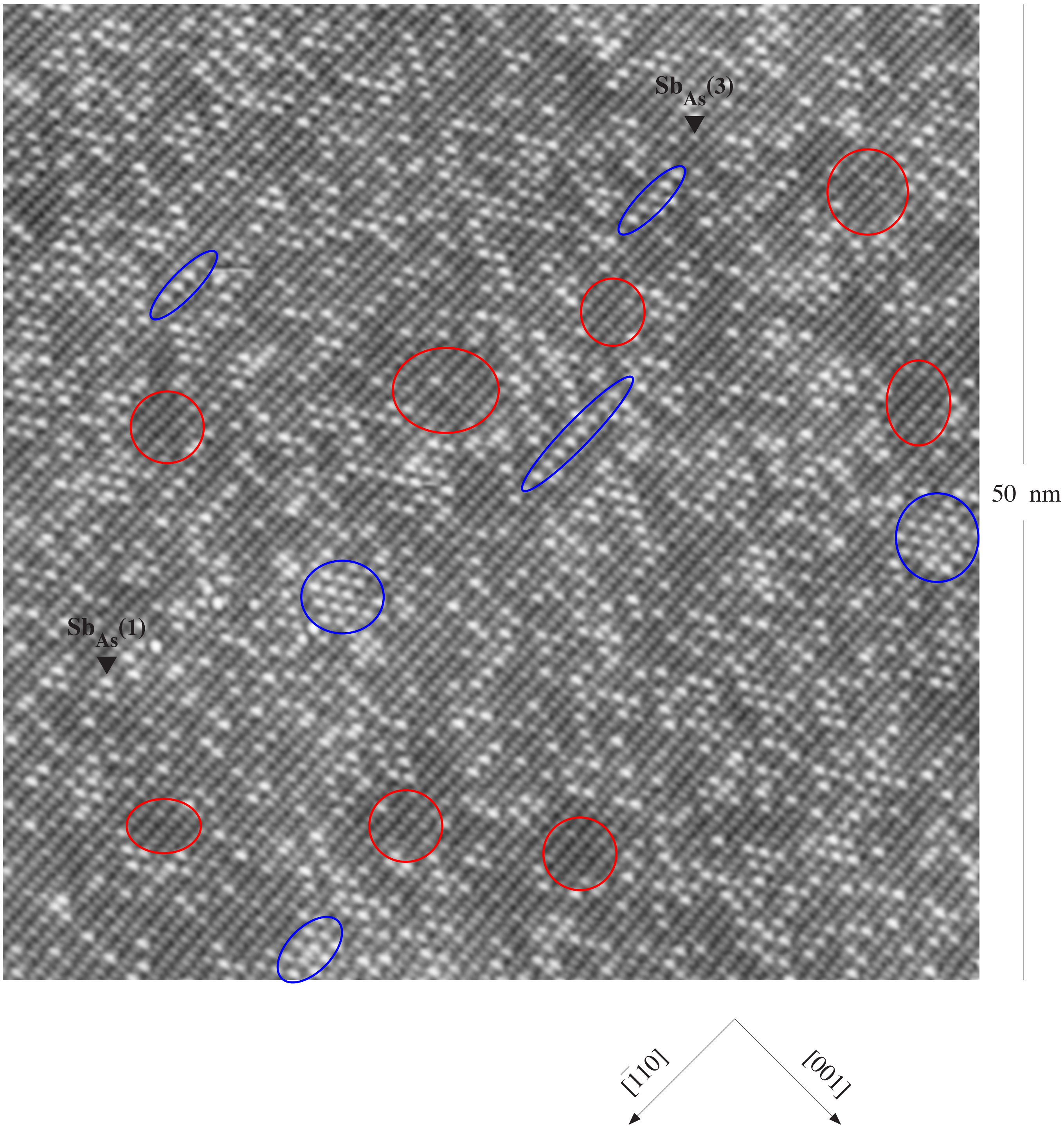

Figure 4

Metamorphic InAsSb

Anion Sublattice Order

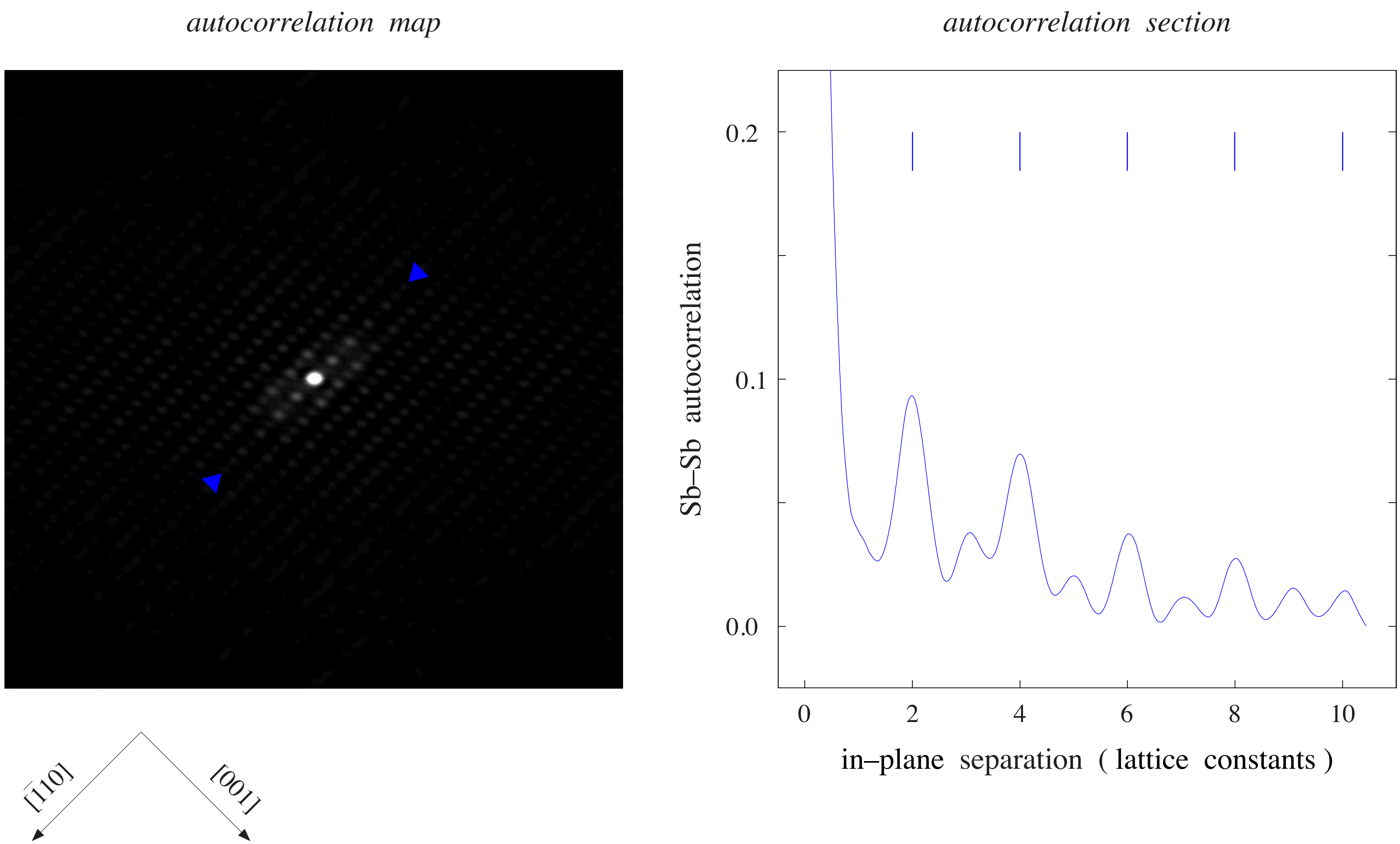

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Figure 5